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(71) Applicant(s)

Rockwool International A/S

(Incorporated in Denmark)

Hovedgaden 501, 2640 Hedehusene, Denmark

(72) Inventor(s)

Jens Gamborg Hansen

(74) Agent and/or Address for Service

Gill Jennings & Every

**Broadgate House, 7 Eldon Street, LONDON,
EC2M 7LH, United Kingdom**

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(54) **Process for the production of saline-soluble vitreous fibres**

(57) A process is provided for producing man-made vitreous fibres having the composition;- SiO₂ 30-70%, Al₂O₃ 0-30%, MgO 2-30%, CaO 10-45%, FeO 0-15%, Na₂O + K₂O 0-10%, P₂O₅ 1-10%, B₂O₃ 0-10%, TiO₂ 0-10%, other oxides 0-15%. In the process a mineral melt is formed by heating charged mineral materials in a furnace and forming the melt into fibres. At least one quarter by weight of the P₂O₅ content of the melt is provided by unrefined ore or industrial waste product having a P₂O₅ content of 2 to 12%. In particular the ore is an unrefined pyroxenite. The ore may be provided as glassy briquettes made by pre-melting and casting the rock.

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ROCKWOOL INTERNATIONAL A/S

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PROCESSES FOR THE PRODUCTION OFMAN-MADE VITREOUS FIBRES

5 This invention relates to improved processes for the production of man-made vitreous fibres (MMVF) of the type commonly known as rock, stone or slag wool fibres.

10 It is well known to form mineral fibres of the type known as rock, stone or slag wool fibres (referred to herein for convenience as stone wool fibres) by melting a charge of mineral materials to form a melt having a desired overall composition and spinning the melt into fibres of the desired analysis.

15 It is generally accepted that modification of the composition of a particular fibre can lead to a change in the physical properties of the fibre. In recent years there has been particular interest in the effect of incorporating phosphorus into the fibres. It has been known for some years that the presence of phosphorus in fibres leads to an increase in the solubility of those
20 fibres, for instance from Uhlman 1978 page 359 to 365; Ceramic Bulletin Volume 5, Number 6, 1978, Ohta, page 602 to 604; Indian Ceramics, July 1968, Mitra, page 97 to 102; Materials Research Society Proceedings, Ed. McVay, volume 26, Plodinek, page 755 to 761; and Glasuren and Ihre Farben
25 1973.

There is no scientific evidence which indicates that stone wool fibres present a health risk, but in recent years manufacturers of stone wool have felt it commercially desirable to provide fibres with improved solubility, which
30 can then be claimed to be of even greater safety than regular stone wool fibres.

Consequently there has been an interest in fibres which contain phosphate. Fibres of this type have been described in for instance EP-A-459,897 and EP-A-558,548.

35 Stone wool fibres generally contain various oxides, for instance silica, calcium oxide, magnesia, alumina, titanium oxide, iron oxides (FeO and Fe_2O_3), sodium oxide,

potassium oxide, manganese oxide, boron oxide and recently, phosphorous oxide.

5 The industry practice when producing stone wool fibres has been to collect together various materials containing varying amounts of the oxides required and to combine these in amounts such that the desired analysis of the melt and fibres can be achieved. Often several different mineral components are required, for instance sometimes up to 8 or even 10 different ingredients.

10 With regard to phosphate-containing fibres, the industry practice has been to take conventional combinations of ingredients and incorporate into these further ingredients which contain a very high concentration of phosphate (for instance 30% or above). For instance in
15 EP-A-459,897, sodium phosphate and calcium phosphate are incorporated into the melt. In EP-A-558,548 it is stated that phosphorus compounds are used.

Other phosphate-containing ingredients which have been used are refined materials produced by treatment of
20 phosphate-containing ores. Such materials include for instance apatite.

These refined materials have a composition which is very different from that of the raw ore from which they are produced. For instance, the amount of phosphate in the
25 refined material can be 5, 8 or even 20 times greater than that of the raw ore. The refinement process also tends to reduce the content of other non-volatile inorganics such as silica, alumina, iron oxides and magnesia.

It has been accepted that such refinement is necessary
30 because it is well known that ores which contain phosphate also tend to have a content of undesirable contaminants. These contaminants include fluoride and other halides, which tend to volatilise in a furnace and escape with the effluent gases into the atmosphere, which is undesirable.
35 A high content of iron is often a characteristic of phosphate-containing ores; this can be undesirable because large amounts of iron can have a corrosive effect in

certain furnaces and on some spinning equipment. For these reasons there is a considerable prejudice against the use of unrefined phosphate-containing ores.

5 Unfortunately, the use of refined phosphate-containing materials has certain disadvantages. These include economic disadvantages; the refined materials are very expensive due to the high costs of carrying out the refining process.

10 Refined phosphate-containing materials tend to be provided in a rather powdery, small particle size form, again as a result of the refining process. This can lead to handling problems during fibre manufacture. In particular, these small particulate materials tend to lead to a high contra pressure in a cupola furnace and give rise to melt flow problems. To overcome such problems it has
15 been found to be essential to use a refined phosphate-containing material such as apatite only by incorporating it, with other particulates, into molded briquettes of particulate mineral material.

20 However, the perceived problems with using raw phosphate-containing ores have meant that until now manufacturers have accepted the disadvantages of using refined phosphate materials as being unavoidable if phosphate-containing fibres are required.

25 It would be desirable to be able to produce phosphate-containing stone wool fibres in an economical and convenient manner, reducing the necessity for use of expensive refined phosphate materials and providing a charge which is simple and convenient to prepare and use.

30 In this specification all percentages are by weight. Elemental analyses are expressed as the oxide and for convenience all iron oxides are expressed as FeO , even though there may be some Fe_2O_3 present also.

35 According to the invention there is provided a process for the production of man-made vitreous fibres having the following composition:

	SiO ₂	30-70%
	Al ₂ O ₃	0-30%
	MgO	2-30%
	CaO	10-45%
5	FeO	0-15%
	Na ₂ O+K ₂ O	0-10%
	P ₂ O ₅	1-10%
	B ₂ O ₃	0-10%
	TiO ₂	0-10%
10	other oxides	0-15%

wherein the process comprises forming a mineral melt by heating a charge of mineral materials in a furnace and forming the melt into fibres,

characterised in that

15 at least one quarter by weight of the phosphate content of the melt is provided by unrefined ore and/or industrial waste product having a phosphate content of 2 to 12%.

20 The process of the invention thus reduces, and in some cases eliminates entirely, the need to use expensive, difficult to handle refined phosphate materials. Instead a significant amount of the charge is made up of a cheaper phosphate-containing raw material which can be introduced in various forms without problems.

25 Suitable unrefined ores having a phosphate content of 2 to 12% for use in the invention include pyroxenites, alkaline basalts and sedimentary rocks which have a suitable phosphate content, for instance sandstone into which phosphate has precipitated from seawater. Preferably
30 the ore contains at least 4%, often around 5 or 6%, phosphate. Generally it contains not more than 10%, often not more than 8 or 9%, phosphate.

Unrefined ores of the defined phosphate content may be used alone or in combination. Particularly suitable ores
35 are those which have an overall composition which is similar to the desired composition of the fibres. When such ores are used it is possible within the process of the

invention to provide a considerable proportion of the charged materials in the form of a single mineral material, which results in increased convenience of manufacture.

Preferred ores have a composition as follows:

5	SiO ₂	30-50%
	Al ₂ O ₃	0.5-8%
	TiO ₂	0.2-4%
	FeO	4-20%
	CaO	15-30%
10	MgO	9-20%
	Na ₂ O	0.1-5%
	K ₂ O	0.1-5%
	P ₂ O ₅	2-9%
	other oxides	up to 10%

15 Suitable ores are naturally occurring apatite-bearing rocks. Particularly preferred ores of which deposits can be found which have such a composition are pyroxenite and related formations. Suitable pyroxenites and other apatite-bearing rocks in alkaline complexes may be found in
20 South Africa, Finland, the Kola peninsula (Russia) and Brazil.

 Suitable unrefined industrial waste products having the required high phosphate content include some converter slags and basic slag from the production of steel from
25 phosphatic iron ores.

 These slags may be obtained as waste products from steel making processes. Any slag having phosphate content as required may be used, but particularly suitable converter slags have a composition as follows:

30	SiO ₂	10-25%
	Al ₂ O ₃	1-2%
	FeO	4-30%
	CaO	45-65%
	MgO	0.5-8%
35	MnO	3-8%
	P ₂ O ₅	2-5%

Other industrial waste products which may be used include waste phosphate-containing stone wool. Use of waste phosphate-containing wool is desirable because it, like preferred unrefined ores, has a composition very similar to that of the fibres which are to be produced from the charge. Its use also involves recycling of waste which is generally desirable, both economically and environmentally. Waste material from fibre production may also be used.

In the process of the invention at least one quarter of the phosphate in the charge is provided by the raw unrefined ore and/or industrial waste material. Mixtures of suitable unrefined ores may be used as may mixtures of suitable unrefined industrial waste materials. The required at least one quarter of the phosphate content of the charge may be provided by a mixture of one or more unrefined ores and one or more unrefined industrial waste products.

In the invention it is preferred that at least one quarter of the phosphate content of the melt is provided by unrefined ores, preferably a single unrefined ore. Unrefined ore materials are preferably present in amounts so as to provide at least 40%, preferably at least 50% of the phosphate content of the melt. Unrefined ores may provide up to 80 or 90% of the phosphate content of the melt although preferably they do not provide more than 70%. For improved economy and simplicity of manufacture it is preferred that these amounts of the phosphate content are each provided by a single unrefined ore.

Industrial waste products, such as converter slag and waste wool and waste material from fibre production processes, may be used, alone or in combination, to provide at least one quarter of the phosphate content of the melt. Generally it is preferred that they are used in combination with unrefined ore material. Converter slag is preferably used in amounts so as to provide up to 20%, preferably up to 15%, more preferably up to 10% of the phosphate content

of the melt, and in combination with waste wool and waste material from fibre production and/or unrefined ore.

Preferably at least 80%, more preferably at least 90% or substantially all of the phosphate content of the melt is provided by a combination of the unrefined ore or ores, converter slag and waste phosphate-containing wool.

Preferably at least one quarter, more preferably at least 40% or 50%, often 60 to 80% although preferably less than 70%, of the entire charge consists of unrefined ore.

Where converter slag is used it is preferably present in amounts of at least 5%, preferably at least 10%, most preferably around 15 to 20%, of the entire charge.

Waste wool may be present in any convenient amount, preferably up to 30%, most preferably 5 to 15%, of the total charge.

The charge of mineral materials may also contain additional ingredients. It is essential that at least one quarter, preferably at least 50%, more preferably at least 70 or 80% of the phosphate in the charge is provided by unrefined ore and/or industrial waste material such as waste wool and converter slag. If necessary additional phosphate may be added in the form of known refined materials. These include apatite, calcium phosphate and sodium phosphate.

Other materials, phosphate-containing or non-phosphate-containing, may be included in the charge in any form to obtain optimum properties of the melt. Suitable components additional to the unrefined ore and/or unrefined industrial waste material may be included in the charge if necessary to achieve the required viscosity and melt temperature. For instance, quartz sand may be included to modify viscosity.

Preferably the melt temperature of the charge is at least 1,000°C, generally at least 1,200°C. Often it is below 1,700°C and is preferably from 1,400 to 1,600°C.

The melt viscosity (measured at 1,400°C) is generally at least 10 or 12 poise, and can be at least 15 poise.

Viscosity is generally below 40 poise, preferably below 30 poise, although it may be as high as 60 or 70 poise.

The charge composition is chosen so as to give the desired fibre analyses. The fibres of the invention have
5 the analysis given above.

A preferred fibre composition is as follows:

	SiO_2	45-60%
	Al_2O_3	0.5-4%
	CaO	10-25%
10	MgO	8-18%
	FeO	5-12%
	$\text{Na}_2\text{O}+\text{K}_2\text{O}$	0-6%
	TiO_2	0.1-4%
	P_2O_5	2-10%
15	other oxides	0-10%

Another preferred fibre composition is as follows:

	SiO_2	32-48%
	Al_2O_3	10-30%
	CaO	10-30%
20	MgO	2-20%
	FeO	2-15%
	$\text{Na}_2\text{O}+\text{K}_2\text{O}$	0-10%
	TiO_2	0-6%
	P_2O_5	1-10%
25	B_2O_3	0-6%
	other oxides	0-15%

Another preferred fibre composition is as follows:

	SiO_2	35-66%
	Al_2O_3	0.5-10%
30	CaO	10-45%
	MgO	2-30%
	FeO	0-10%
	$\text{Na}_2\text{O}+\text{K}_2\text{O}$	0-7%
	TiO_2	0-10%
35	P_2O_5	2-10%
	B_2O_3	0-10%
	other oxides	0-15%

The process of the invention may be carried out using any furnace suitable for the production of stone wool fibres. Preferably the furnace is one which is designed such that the charged mineral materials form a gas permeable column in the furnace and the melt is formed at the base of the column. Thus preferably the furnace has an inlet at or near its top for receiving the charge, at least one gas inlet at or near its base for blowing a gas inlet stream into the furnace, a gas outlet at or near its top for the escape of gas effluent and is arranged to hold a column, for instance at least 1 metre high, of unmolten charged mineral material above the melt which is at the base of the furnace. In such a furnace the column of charge gradually melts and drips down into the pool of melt at the base of the furnace and is continuously replenished from above.

The furnace may be heated in any suitable manner, for instance by electrical heating, oil or gas heating or by combustion of solid carbonaceous material.

Preferably the furnace is a shaft furnace, more preferably a cupola furnace, although tank furnaces may be used.

We find surprisingly that if a furnace such as that described above containing a column of unmolten charged material is used then significantly lower amounts of halide effluent gases are emitted from the gas outlet than would be expected of a halide-containing unrefined phosphate ore.

Any halide or SO_2 gases which are emitted may be cleaned up using known methods, for instance by absorbing onto a suitable absorbent such as chalk, lime, limestone or magnesia.

A preferred system for dealing with fluoride-containing raw materials is described in our copending application GB 9513363.3, filed 30 June 1995, not yet published.

The melt may be formed into stone wool fibres in any conventional manner. For instance it can be poured into a

cup which spins about a substantially vertical axis and which has perforations in its walls out through which the melt is extruded and then thrown as fibres.

5 The jet blowing process (known as Düsen Blasen Verfahren) may be used.

10 A preferred fiberisation method comprises pouring the melt onto the outer surface of one or more rotors, each mounted about a substantially horizontal axis, from which the melt can be thrown as fibres. Preferably the rotors are arranged in a cascade formation, so that melt poured onto the top rotor is thrown from the rotor partially as fibres and the remainder onto a second, subsequent, rotor, from which more fibres are thrown. If desired, two and especially three subsequent rotors may be used, giving a
15 total of four rotors in the cascade.

When a phosphate-containing ore is used in the process of the invention it is unrefined. This is in contrast to the refined materials such as apatite which have been used up until now. Refining processes are designed so as to
20 increase the concentration of phosphate in the material and considerably reduce the concentration of other non-volatile inorganics such as silica and alumina. Thus in the invention an unrefined ore is one which retains essentially the same proportions of non-volatile inorganics as in its
25 natural form. The unrefined ore may be added to the furnace in the form of crushed rock. Alternatively it may be converted to fine particulate form and formed into briquettes, optionally together with other particulate mineral material and binder.

30 Unrefined industrial waste products may also be provided in any convenient form. They may be converted to particulate form and incorporated into conventional briquettes of particulate mineral material, either alone to form briquettes consisting essentially only of the
35 industrial waste product or as a mixture with other mineral materials. They may be incorporated in briquettes of

particulate mineral material together with particulate unrefined ore. Waste wool is preferably incorporated in briquettes.

Industrial waste materials may be incorporated into
5 the charge as received if this is convenient.

In the invention it is particularly preferred to provide the unrefined ore in the form of glassy briquettes which have been formed by pre-melting the ore in a furnace and casting the molten ore into briquette form. These
10 glassy briquettes may then be added directly to the furnace as part of the charge of mineral materials.

The glassy briquettes may consist substantially only of the unrefined ore. Alternatively, additional components of the final charge may be melted in the pre-melting
15 furnace to form glassy briquettes which comprise unrefined ore and other charge components. If convenient, all components of the charge may be provided in the form of glassy briquettes formed by pre-melting. This may be in the form of mixed briquettes, each containing several
20 components of the charge or in the form of differing briquettes each consisting substantially only of a single charge component in cast form.

Pre-melting is carried out in a pre-melting furnace separate from the fibre-forming furnace. The pre-melting
25 furnace may be any suitable furnace. It can be for instance a rotary kiln but is preferably a tank furnace. Heating may be by any convenient method, but preferably it is electrical.

In the pre-melting furnace the unrefined ore and other
30 charge components if any are heated to a temperature of at least 1,200°C and usually below 1,700°C. When molten they are cast into briquettes.

A particular advantage of the pre-melting process is that it is a simple and convenient method for transforming
35 raw unrefined ore into an easily transportable form. The briquette form also gives rise to fewer dusting problems than does ore in the form of rocks. It allows pre-melting

furnaces to be used on or near the excavation site to convert the ores mined into an easily transportable form, for transfer to the site where fibre-formation is carried out.

5 A further advantage is that the pre-melting process can be designed to induce volatilisation of unwanted contaminants such as halides and SO_2 gas-forming components which escape from the ore on heating. These unwanted contaminants are thus cleaned up during the pre-melting
10 process and not carried into the fibre-forming furnace. This is particularly advantageous for phosphate-containing pyroxenites, which tend to contain halogen contaminants. It is also advantageous for instance sedimentary rocks which also tend to have a significant content of halides.

15

Although the pre-melting process is particularly useful in the context of the present invention, the above advantages also apply where any unrefined ore is being used, whether phosphate-containing or not.

20 Thus according to a second aspect of the invention there is provided a process of forming glassy briquettes by melting at a temperature of from 1,200 to 1,700°C unrefined ore or unrefined industrial waste products.

Also according to a third aspect of the invention
25 there is provided a process for the production of man-made vitreous fibres having the composition:

	SiO_2	30-70%
	Al_2O_3	0-30%
	MgO	2-30%
30	CaO	10-45%
	FeO	0-15%
	$\text{Na}_2\text{O}+\text{K}_2\text{O}$	0-10%
	P_2O_5	1-10%
	B_2O_3	0-10%
35	TiO_2	0-10%
	other oxides	0-15%

wherein the process comprises forming a mineral melt by heating a charge of mineral materials in a furnace and forming the melt into fibres, characterised in that at least 10% by weight of the charge is provided by glassy
5 briquettes produced according to the second aspect of the invention.

The processes of the second and third aspects of the invention are particularly advantageous when the unrefined ore or industrial waste product has a content of volatile
10 contaminant, such as halogens. The process may however be used for converting into conveniently transportable form any naturally occurring ore.

The fibres formed according to any of the processes of the present invention may be used for any of the purposes
15 known for MMV fibres, for instance slabs, sheets, tubes and other shaped products for thermal insulation, fire insulation and protection, noise reduction and regulation. They may also be used as horticultural growing media or, as free fibres, as reinforcements for cement, plastics and
20 other materials or as filler.

The following is an example of the invention.

Example 1

Charges C to G as described below are used to form a melt in a cupola furnace and to form fibres. Compositions
25 C to G are charges according to the invention.

		C	D	E	F	G
Charge (weight%)	Pyroxenite	20.0	40.0	60.0	80.0	95.0
	Converter Slag	4.8	5.3	5.0	0.0	0.0
	Briquette (1)	37.6	54.8	35.0	20.0	5.0
	Briquette (2)	37.6	0.0	0.0	0.0	0.0
Briquette (1) (weight%)	Cement	11.0	11.0	11.0	11.0	11.0
	Apatite sand	13.3	4.4	0.5	0.0	0.0
	Quartz sand	32.7	35.7	42.0	42.0	40.0
	Olivine sand	28.5	12.7	0.0	0.0	0.0
	Wool waste	14.5	28.0	35.5	27.0	20.0
	Iron ore	0.0	8.2	11.0	20.0	29.0
Briquette (2) (weight%)	Cement	10.9				
	Iron ore	10.7				
	Converter slag	14.9				
	Quartz sand	38.8				
	Wool waste	13.3				
	Olivine sand	11.4				
Fibre chemistry (weight%)	SiO ₂	53.5	53.4	52.6	5.8	48.2
	Al ₂ O ₃	1.3	1.5	1.7	1.9	2.0
	TiO ₂	0.2	0.2	0.3	0.3	0.3
	FeO	6.1	6.0	5.7	5.2	3.9
	CaO	20.6	20.6	22.1	21.7	23.2
	MgO	12.6	12.2	11.2	13.1	14.5
	Na ₂ O	0.2	0.3	0.3	0.3	0.3
	K ₂ O	0.4	0.7	1.0	1.2	1.4
	MnO	0.2	0.2	0.1	0.0	0.0
	P ₂ O ₅	4.0	4.0	4.0	4.6	5.2
	Melt viscosity at 1,400°C (Poise)	14.2	15.0	14.6	12.6	10.1

The pyroxenite is an ore mined in South Africa and has the following composition:

5	SiO ₂	46.4%
	Al ₂ O ₃	2.0%
	TiO ₂	0.3%
	FeO	4.4%
	CaO	23.1%
10	MgO	14.6%
	Na ₂ O	0.3%
	K ₂ O	1.4%
	P ₂ O ₅	5.2%

The converter slag has the following composition:

15	SiO ₂	11.8%
	Al ₂ O ₃	1.2%
	TiO ₂	0.6%
	FeO	21.2%
	CaO	49.9%
20	MgO	0.9%
	Na ₂ O	0.3%
	K ₂ O	less than 0.1%
	P ₂ O ₅	2.6%
	MnO	2.2%

CLAIMS

1. A process for the production of man-made vitreous fibres having the following composition:

	SiO ₂	30-70%
5	Al ₂ O ₃	0-30%
	MgO	2-30%
	CaO	10-45%
	FeO	0-15%
	Na ₂ O+K ₂ O	0-10%
10	P ₂ O ₅	1-10%
	B ₂ O ₃	0-10%
	TiO ₂	0-10%
	other oxides	0-15%

wherein the process comprises forming a mineral melt
15 by heating charged mineral materials in a furnace and forming the melt into fibres, characterised in that

at least one quarter by weight of the phosphate content of the melt is provided by unrefined ore or industrial waste product having a phosphate content of 2 to
20 12%.

2. A process according to claim 1, in which at least half by weight of the phosphate content of the melt is provided by the unrefined ore or industrial waste product.

3. A process according to claim 1 or claim 2 in which one
25 quarter to 70% of the total melt is provided by the unrefined ore or industrial waste product.

4. A process according to any preceding claim in which at least half by weight of the phosphate content of the melt is provided by a naturally occurring rock, preferably an
30 unrefined pyroxenite ore having a phosphate content of 3 to 10% or a sedimentary rock having a phosphate content of 3 to 10%.

5. A process according to any preceding claim in which the unrefined ore is pyroxenite ore or sedimentary rock and
35 is provided in the form of glassy briquettes which have been made by pre-melting the rock and casting it into briquettes.

6. A process in according to any preceding claim in which the unrefined ore is pyroxenite ore provided in the form of crushed rock.

5 7. A process according to any preceding claim in which the furnace has an inlet at or near its top for receiving the charged mineral materials, at least one gas inlet at or near its base for blowing a gas inlet stream into the furnace, a gas outlet at or near its top for escape of gas effluent

10 and is arranged to hold a column of unmolten charged mineral materials above the melt which is at the base of the furnace.

8. A process according to any preceding claim in which the furnace is a cupola furnace.



Application No: GB 9516759.9
Claims searched: 1-8

Examiner: C.A. Clarke
Date of search: 29 October 1996

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.O): C1M(MAL)

Int Cl (Ed.6): C03C 13/00,13/06

Other: ONLINE: WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X,E	WO 95/21799 A ROCKWOOL see examples and p7 line 29	1-4 at least

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.
& Member of the same patent family

A Document indicating technological background and/or state of the art.
P Document published on or after the declared priority date but before the filing date of this invention.
E Patent document published on or after, but with priority date earlier than, the filing date of this application.